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METHODOLOGY FOR CERAMIC LIFE PREDICTION AND RELATED PROOF TESTING

INTERIM REPORT FOR THE PERIOD
JANUARY 1977 — MARCH 1978

R. K. Govila

FORD MOTOR COMPANY
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Prepared for
ARMY MATERIALS AND MECHANICS RESEARCH CENTER
Watertown, Massachusetts 02172

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FOREWORD

This report is the first interim technical report of the "Methodology For Ceramic Life Prediction" program initiated by the Energy Research and Development Administration (ERDA), now Department of Energy (DOE) and monitored by the Army Materials and Mechanics Research Center, under Contract Number DAAG-46-77-C-0028. This work is necessary in formulating a methodology for ceramic life prediction using the material property characterization data so that ceramic materials can be used in high temperature structural applications.

Since this work is continuing, all strength characterization property data contained in this report must be considered tentative and the report should be considered to be illustrative of establishing a methodology for predicting the lifetime reliability of structural ceramic materials in high temperature applications.

The principal investigator of this program is Dr. R. K. Govila, Ford Motor Company, and the technical monitor is Dr. E. M. Lenoe, AMMRC. The author is thankful to the following people for the valuable contributions made by them in the preparation of this report: C. Knapp for designing the load train set-up, R. M. Williams and J. Uy for flexure stress-rate data, R. Elder for stress-rupture data, A. Paluszny for proof test methodology, R. Predmesky, P. Cherep, and G. Grab for polishing and testing of MOR specimens. Thanks are also due to J. R. Secord, A. F. McLean and C. L. Magee for encouragement throughout the work. Finally, it is a pleasure to thank Dr. E. M. Lenoe, AMMRC for valuable suggestions in carrying out the program and to Mr. R. B. Shulz of DOE for supporting the program.

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ABSTRACT

This program consists of a basic study using two potential high temperature ceramic materials, namely, hot pressed silicon nitride NC132 (Norton) and hot pressed silicon nitride made with 3.5 wt. percent MgO (Ford material) to determine the statistical and time dependent strength characteristics related to the presence of subcritical crack growth. The data will be used to correlate analytical ceramic life prediction and possibly verify with experimental results obtained from simple tensile stress rupture tests.

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1. INTRODUCTION

High temperature failure of ceramic materials which are made by hot pressing methods is usually influenced by the presence of subcritical crack growth (SCG). In this mechanism, the randomly distributed inherent flaws in the material grow to some critical size and thus lead to catastrophic failure. Therefore, this study consists primarily of a basic investigation using two potential high temperature structural ceramic materials, namely, hot pressed silicon nitride NC-132 (Norton Co., Mass.) and hot pressed silicon nitride made with 3.5 wt. percent MgO (Ford material designated as FHPSN) to determine the statistical and time-dependent strength characteristics related to SCG and to use this data as a fundamental basis for correlating analytical life predictions in simple geometric shapes. The program will further investigate room temperature fast fracture testing methods as a means of proof-testing for long time evaluation. The established methodology for ceramic turbine materials would then be used to analytically determine a proof test for duo-density silicon nitride (FHPSN hub) turbine rotors and predict resulting rotor life and reliability. In summary, the program will investigate and evaluate the following parameters for both type of materials:

(i) Determine the inherent flaw size, a_o, using indentation induced flaw technique.

(ii) Establish the temperature for the onset of SCG using indentation induced flaw technique.

(iii) Determine the Critical Stress Intensity Factor, K_{IC}, using the double torsion method.

(iv) Determine crack velocity, V, the corresponding stress intensity factor, K_I, and the associated fracture mechanics parameters A and 'n' for subcritical crack growth, using the double torsion method.

(v) Conduct detailed four-point testing of bend bar specimens at four different temperatures up to 1371°C (2500°F) at three different machine head speeds (strain rates). From the test results, determine Weibull characteristic strength, σ_θ , and modulus, m, and crack propagation parameter 'n'.

(vi) Determine the strain rate dependence of NC-132 as a function of temperature using indentation induced flaw technique.

(vii) Analytically predict time to failure, t_f, based on strength parameters as determined using flexure stress rate measurements (v) and compare with that as predicted by the fracture mechanics approach using double torsion method.

(viii) Conduct tensile stress rupture testing of NC-132 material to determine time to failure, t_f, for a given applied stress and temperature.

(ix) Correlate the actual time to failure from the tensile stress rupture testing with that of analytical predictions based on stress rate approach.

2.0 FRACTURE MECHANICS APPROACH

2.1 Inherent Flaw Size Determination

In this study, the indentation induced flaw (or controlled flaw) technique has been used to estimate the inherent flaw size in hot pressed silicon nitride NC-132.

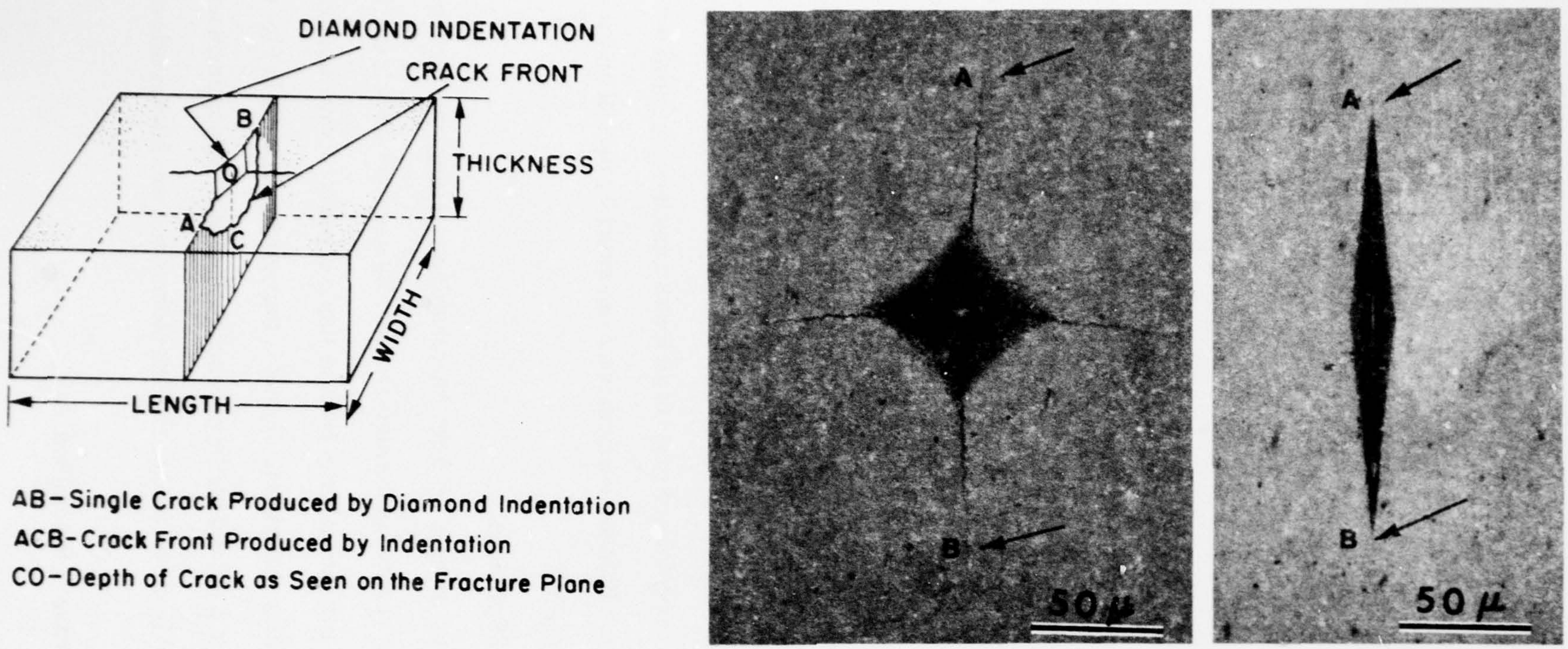
2.1.1 Sample Preparation and Testing

Test specimens of dimensions 1.25 in. (~ 32 mm) long x 0.25 in. (~ 6.5 mm) wide x 0.125 in. (~ 3.5 mm) thick were machined from blocks of material such that the tensile face was perpendicular to the hot pressing direction (strong direction). All faces were polished in a lengthwise direction using 220-grit diamond wheels and the edges chamfered to prevent notch effects. One surface (the tension face in bending) was carefully ground and wet polished to 6 μ diamond finish. The specimens were then precracked using the indentation technique as outlined briefly in Section 2.1.2, and tested in four-point bending in an Instron machine at a constant crosshead speed of 0.005 in./min. (0.127 mm/min.). The outer and inner knife edges were 0.75 in. (~ 19 mm) and 0.375 in. (~ 9.5 mm) span respectively. The high temperature bend tests were performed in air in an Instron machine equipped with a rapid temperature furnace.* Fracture surfaces were examined by optical and scanning electron microscopy (SEM).

2.1.2 Controlled Precracking of Specimen

Indenting the specimens using a Vickers microhardness testing machine with diamond pyramid indenter resulted in the most satisfactory and reproducible cracks. This technique has been used successfully both in single crystals of vanadium carbide [1] and in polycrystalline materials [2-5], and is shown schematically in Fig. 1a. Typical example of a crack produced on the polished surface of a test specimen with 4000 gm. indentation load is shown in Fig. 1b. The indentation was always oriented

*CM Inc., High Temperature Furnaces, Bloomfield, N.J.



a.   b. 4000 gm   c. 2600 gm

Fig. 1(a) Schematic representation of crack geometry in a specimen

Fig. 1(b) Typical example of a crack produced on the polished surface of hot pressed Si_3N_4 (NC132) test specimen using 4000 gm. indentation load.

such that the crack AB or crack front ACB, Fig. 1, was always perpendicular to the maximum tensile fiber stress. The crack depth, CO, was measured directly from the micrographs of fracture face. Cracks can be produced with indentation loads as low as 500 gm. but repropagation of such cracks was not always successful because the inherent flaws in the material were about the same size. Therefore, the lowest indentation load used operationally in this study was 1000 gm. At indentation loads up to 4000 gm. the crack fronts were approximately semi-circular. All indentations were made at room temperature.

2.1.3 Influence of Crack Size on Fracture Stress at Room Temperature

A series of specimen was prepared and precracked with 1000 gm., 2000 gm., and 4000 gm. indentation loads. The variation of fracture stress, σ_F, with crack length (actually inverse square root of crack depth) for hot pressed silicon nitride NC-132 specimen tested at room temperature is shown in Fig. 2. Although, there is some scatter in the data, a reasonably good linear relationship is evident which correlates with the Griffith [6] flaw criterion for brittle fracture. However, from a practical point of view, perhaps the most useful application of the data in Fig. 2 is as a means of estimating the inherent flaw size in this material. Extrapolation of the data in Fig. 2 to the fracture stress of uncracked samples implies that fracture initiating flaws of the order of 10 to 20 microns in size are present in virgin hot pressed silicon nitride NC-132. In conclusion, the inherent flaw size, a_o, in the as received (virgin) hot pressed silicon nitride NC-132 material is in the order of 10 to 20 microns.

With respect to future work, similar studies will be made on 3.5% MgO HPSN (Ford) material to evaluate the flaw size, a_o.

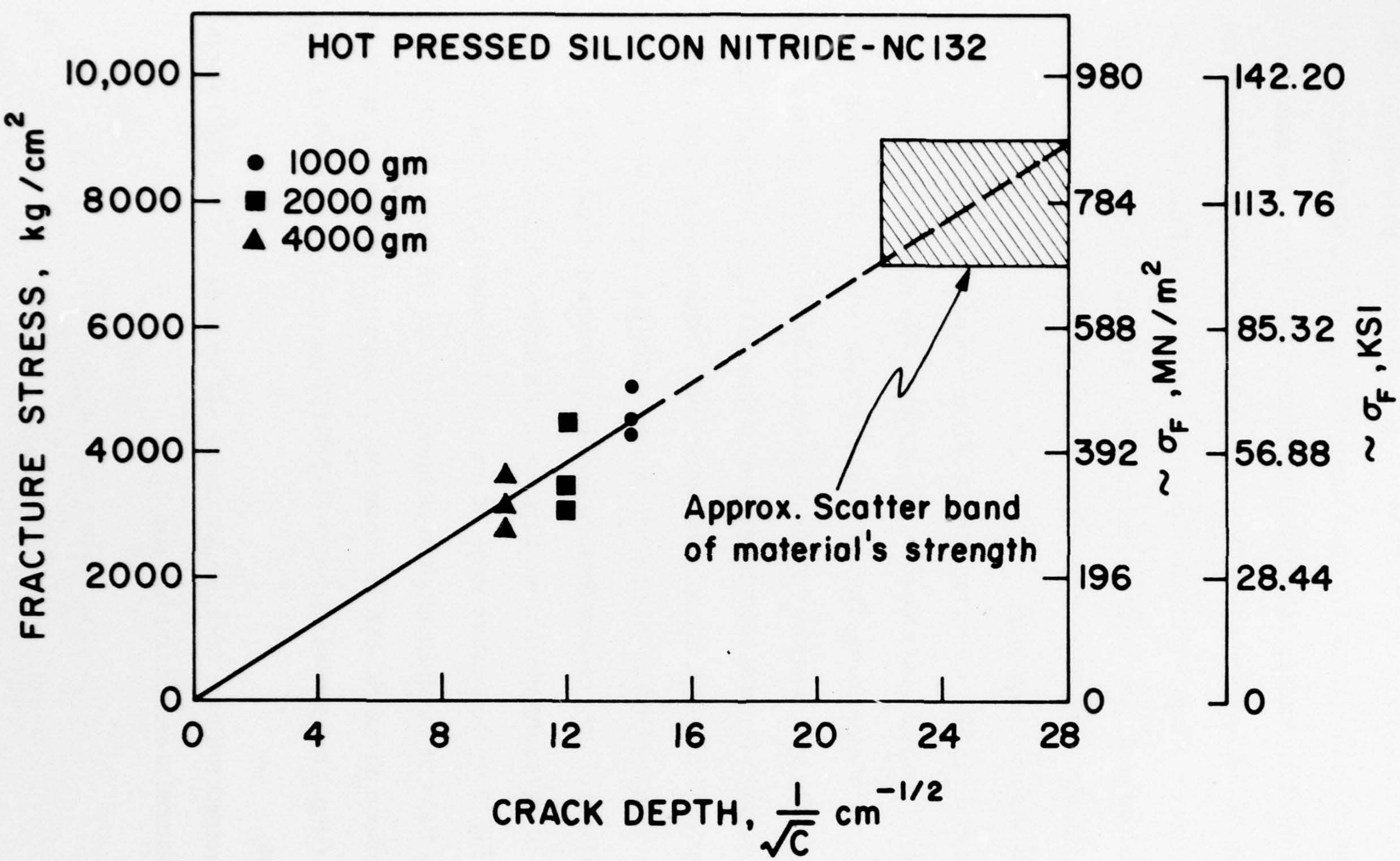


Fig. 2 Variation of fracture stress as a function of inverse square root of crack depth at room temperature.

2.2 Nominal Fracture Toughness Determinations

In this study, the double torsion (DT) method as outlined by Evans [7] and Williams and Evans [8], and the indentation induced flaw technique [1-5], have been used to evaluate both the nominal fracture toughness, K_{IC} (or the critical stress intensity factor) for catastrophic failure and subcritical crack growth parameters in hot pressed silicon nitride NC-132 as described briefly below:

2.2.1 Double Torsion Method

2.2.1.1 Sample Preparation, Testing and Analysis

The DT specimens used were approximately 3 in. ($\sim$ 75 mm) long, 1 in. ($\sim$ 25 mm) wide and 0.050 in. ($\sim$ 1.25 mm) thick. A schematic of the DT specimen and loading configuration is shown in Fig. 3. A prerequisite of the DT test is the presence of a precrack in the test specimen with a crack length of approximately one-half the width of the specimen. For crack velocity and corresponding stress intensity measurements, it is the precrack which is to be repropagated at test temperature. Normally, a very fine notch or slit is made in the center of a DT specimen in order to facilitate the initiation of a precrack when loaded in the double torsion fixture. The notch or slit is usually accompanied by a narrow groove along the central length of the specimen to guide crack propagation. The groove depth was varied systematically from 0.005 in. to 0.025 in. The double torsion testing fixture used, Fig. 3, was similar in design to that used by Evans and Wiederhorn [9] and made from hot-pressed silicon carbide. The stress intensity factor, K_I, for a DT specimen of an elastic material under plane stress conditions is given by:

$$K_I = P\, W_m \left[\frac{3(1+\nu)}{W\, d^3\, d_n}\right]^{1/2} \quad \text{--------(1)}$$

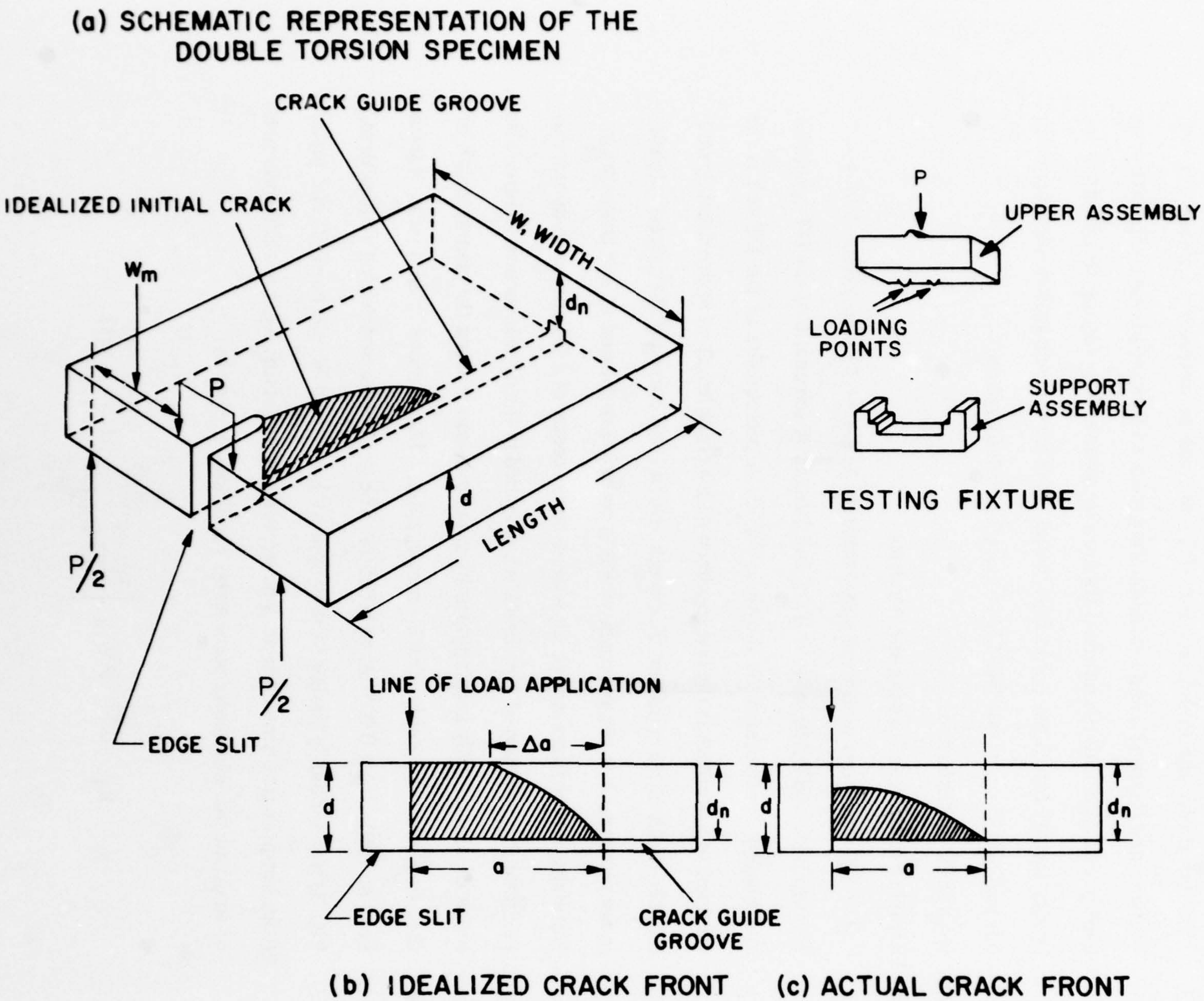


Fig. 3 Schematic display of the Double Torsion Specimen

Where P is the applied load, ν is Poisson's ratio, W_m is the moment arm, W is the specimen width, d is the specimen thickness and d_n is the specimen thickness in the plane of the crack or net thickness. Specimen dimensions are shown in Fig. 3. Note that K_I depends only on the applied load P, specimen dimensions, and Poisson's ratio and is independent of crack length according to elementary theory. It is this feature that makes the DT test* very popular for making subcritical crack growth studies.

The stress intensity-crack velocity data were obtained by the load-relaxation method. In this, a sharp crack is initiated, the DT specimen is reloaded in the testing fixture in the Instron testing machine at a fast speed, the machine crosshead is stopped and the load-relaxation recorded on the chart recorder. Following the work of Evans [7] and others [8-11], the crack velocity ($V = \frac{da}{dt}$) at a particular load P is computed from the rate of load relaxation (at constant displacement, $\frac{dy}{dt} = 0$) is given by:

$$V = \frac{da}{dt} = -\phi \frac{P_{i,f}}{P^2} \left(a_{i,f} + \frac{C}{B}\right) \left(\frac{dP}{dt}\right)_y \quad \text{-------} (2)$$

Where $P_{i,f}$ is the initial (i) or final (f) load, P is the instantaneous load, $a_{i,f}$ is the initial or final crack length and B and C are constants relating to the slope and intercept of a compliance calibration analysis of the DT specimen, y is the elastic specimen deflection and ϕ is a geometrical factor relating to crack front profile [7].

*Some of the difficulties encountered in making a successful DT test are described in Sec. 2.2.1.2.

Assuming that the initial crack is large and contributions due to various constants (as indicated in equation 2) are small, the crack velocity expression can be further simplified and given as follows:

$$V = -\frac{P_{i,f} \quad a_{i,f}}{P^2} \left(\frac{dP}{dt}\right)_y \quad \text{--------} (3)$$

In general, the following method was used for measuring K_I and V from the DT specimen:

(a) After placing the DT specimen in the test fixture, it is loaded in the Instron machine at a slow crosshead speed of 0.001 in./min. at room temperature (or higher temperature as specified) in air. The Instron machine is kept running until a sudden load drop occurs indicating the nucleation or pop-in of a precrack and immediately the crack is arrested by stopping the machine crosshead and the specimen is unloaded at a fast crosshead speed. The corresponding loads for crack pop-in and crack arrest are designated P_O and P_A respectively. When the initial crack is nucleated at a higher temperature such as 1300°C or 1400°C, the load versus deflection/time curve does not show a sudden load drop but simply rounds off at the maximum load and afterwards a rapid decrease in load occurs indicating the nucleation of a crack.

(b) For K_{IC} determination, the specimen is reloaded in the Instron machine at a fast crosshead speed of 0.1 in./min. or 0.5 in./min. at the desired test temperature and the fracture load, P_c, for catastrophic crack propagation is measured. This load is usually slightly smaller than the load P_O*.

*If the crack tip is blunted due to accompanying plastic deformation or the moving crack is slowed down by the presence of cleavage steps, it is likely that P_c will be greater than P_O.

(c) For crack velocity measurements, the specimen containing the initial crack is reloaded at a fast crosshead speed of 0.1 in./min. to some load P such that $P_A < P < P_O$, at the desired test temperature and the crosshead stopped to get the load-relaxation curve. Using equation 3, and the load-relaxation curve, the crack velocity V can be calculated.

2.2.1.2 Nominal Fracture Toughness, K_{IC}, Evaluation

Values of K_{IC} for hot pressed silicon nitride NC-132 at 20°C using equation (1) are given in Table 1, and vary from 3.9 to 4.4 $MN/m^{3/2}$. The average value of K_{IC} is about 4.1 $MN/m^{3/2}$. Evans and Widerhorn [9] using this technique in a similar type of material, hot pressed silicon nitride HS130, found K_{IC} at 20°C to vary from 4.2 to 5.3 $MN/m^{3/2}$ with an average value of 4.7 $MN/m^{3/2}$. Typical fracture surface of a DT specimen in which the initial crack is produced at 20°C and subsequently repropagated at the same temperature (20°C) at a fast speed is shown in Fig. 4. Note that this specimen contained a central groove (about 0.005 in. deep) for guiding the crack straight. However, machining the groove caused the nucleation of cleavage steps (as shown by small vertical arrows, Fig. 4) all along the length of the groove. Therefore, it is possible that these cleavage steps could blunt the crack tip of the initial crack and may increase the required critical load, P_c, for fast fracture.

The DT test may appear simple but in our work considerable difficulties were encountered as summarized briefly below:

(i) The initiation of precrack at 20°C often does not occur inside the leading groove but occurs tangential to it. Upon repropagation, the initial crack (precrack) runs out to the side instead of following the straight path. Those specimens in which the crack ran or curved out to the side were considered as an unsuccessful test. A successful DT test is that in which the initial crack upon

TABLE 1. Fracture Toughness Measurements in Hot Pressed Silicon Nitride NC132 at 20°C and at constant Crosshead Speed

Specimen No.	Grooved Specimen: Yes, Groove Depth	Grooved Specimen: No	Precracking: Initiation Temp. °C	Precracking: Initial Length mm	Precracking: Repropagation Temp. °C	Fast Fracture Load P_c pounds	Stress Intensity Factor, MN/m$^{3/2}$: K_I	Stress Intensity Factor, MN/m$^{3/2}$: K_{IC}	Remarks
3 A		X	20	20	20	16		3.9	Crack repropagated centrally all through the length
4 A		X	"	9	"	18		4.4	"
5 A		X	"	12	"	17		4.15	"
10 A	X 0.127 mm		"	15	"	16		4.06	"
8 B		X	1350	16	"	28	6.63		"
11 C	X 0.127 mm		20	8	1400	31	7.87		Load-Relaxation curve obtained
21 D	X 0.315 mm		1350	-	1350	23	6.4		"

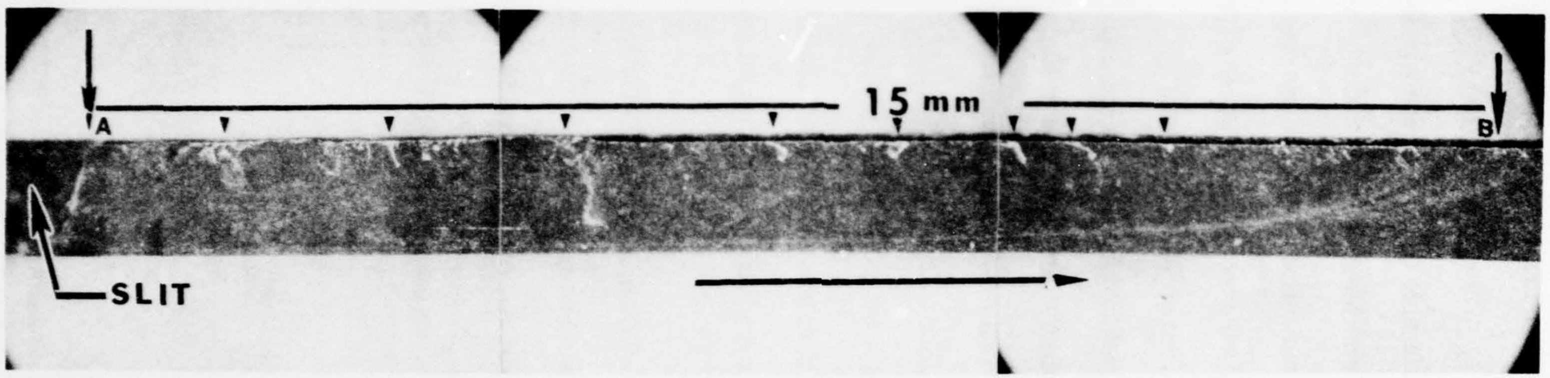


Fig. 4 Typical fracture surface of a double torsion specimen in which the initial crack AB (precracked) was produced at 20°C and subsequently repropagated at 20°C. Cleavage steps (as marked by short vertical arrows) are visible all along the machined groove length. Horizontal arrow indicated the direction of crack propagation.

repropagation travelled centrally all through the length of the specimen.

(ii) It would be preferable to have no lead groove in the specimen as grooving produces cleavage steps all along the crack path and these cleavage steps may slow down the moving crack. However, this would require great experimental care in alignment and large number of samples if no groove were used.

(iii) Initial cracks produced at room temperature appear to have crack fronts sharper than those produced at higher temperatures (1300-1400°C). This conclusion is based on preliminary data and further work has to be done to investigate this completely.

(iv) Often during the load-relaxation curve, the moving crack gets blunted at the tip either due to plastic deformation or crack branching which then leads to an unsuccessful test.

The data shown in Table 1 is obtained from a total of 25 DT tests showing the large number of unsuccessful tests which were encountered.

In conclusion, the average value of K_{IC} for hot pressed silicon nitride NC-132 using double torsion technique at 20°C is 4.1 $MN/m^{3/2}$. Future work will include similar studies to determine K_{IC} for 3.5% MgO HPSN (Ford) rotor hub material.

2.2.2 K_{IC} Determination by Indentation Induced Flaw Technique

The fracture mechanics approach as used by Petrovic et. al. [5] was used to determine the critical stress intensity factor, K_{IC}, from the knowledge of flaw dimensions and the corresponding magnitudes of fracture stresses of precracked specimens at room temperature. The value of K_{IC} for NC132-Si_3N_4 is about 3.5 $MN/m^{3/2}$ at 20°C and is comparable to the value of 4.1 $MN/m^{3/2}$ as determined by the double torsion method (see Sec. 2.2.1.1). The small difference in magnitudes of K_{IC} values as determined by two different methods, namely, the Indentation Induced Flaw Technique and the Double Torsion is primarily due to the fact that the former is a "micro" and the latter is a "macro" method respectively. Future work will also determine K_I as a function of temperature.

In conclusion, use of the indentation induced flaw technique resluted in a K_{IC} at 20°C for NC132 is about 3.5 $MN/m^{3/2}$.

2.3 Crack Velocity Determination

A functional relationship between crack velocity (V) and the corresponding stress intensity factor, K_I, for subcritical crack growth has been approximately described by the following relationship;

$$V = A K_I^{\,n} \text{-------} (4)$$

where A and n are constants. This is the first formulations to be used in proof test methodology and failure prediction [illegible] ic turbine engine components. The procedure used for measuring V [illegible] $_I$ has been outlined in Sec. 2.2.1.1. Two successful DT tests have been made at 1400°C and 1350°C as indicated in Table 1. Typical fracture surface of a specimen for which the load-relaxation curve is obtained at 1400°C is shown in Fig. 5. Future work will be directed in calculating the V,

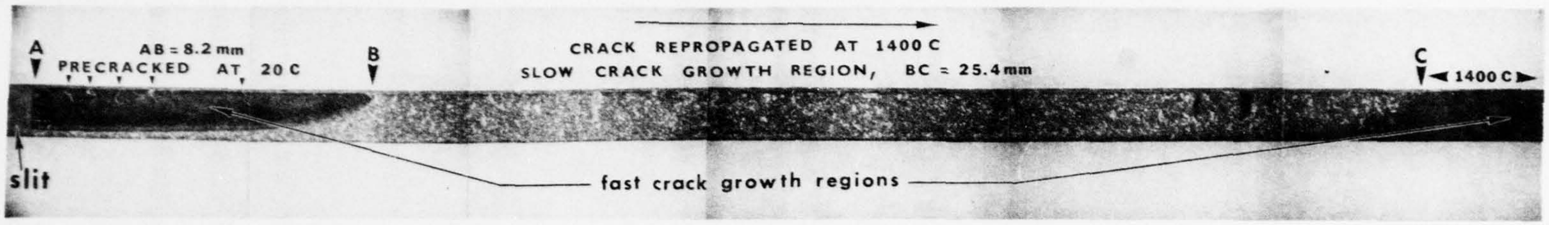


Fig. 5 Typical fracture surface of a double torsion specimen in which the initial crack AB produced at 20°C and subsequently repropagated at 1400°C. A load-relaxation curve is obtained on this specimen.

K, A and n parameters from these load-relaxation curves.

2.4 Temperature Dependence of Fracture Stress Using Indentation Induced Flaw Technique

The temperature dependence of the fracture stress, σ_F , for uncracked (virgin) material (NC132) and for specimens precracked with a Diamond Pyramid Indenter (4 Kg. load indentation produced cracks of about 95 $\pm$ 5 micron deep) and Knoop Indenter (2600 gm. load indentation produced cracks of about 83 $\pm$ 2 micron deep), and tested at temperatures between 20 and 1400°C in air is shown in Fig. 6*. It is clear that σ_F for precracked specimens remains essentially constant and independent of temperature from 20 to 1000°C. The constancy of σ_F indicates that little or no blunting of the crack tip by plastic deformation occurred in this temperature range. Typical fracture surfaces of specimens tested at 20 and 1000°C are shown in Fig. 7. Note that the mode of fracture in the precracked region (inside ACB) and in the repropagated region (outside ACB) is similar i.e. mixed mode of fracture consisting of transgranular and intergranular crack growth, Fig. 7. The sudden and subsequent increase in σ_F at temperatures between 1000-1250°C (Fig. 6) is interpreted as indicative of blunting of the microcrack by plastic flow at the tip of the crack. At temperatures above 1250°C, subcritical crack growth starts and the σ_F curves for virgin and precracked specimens merge together followed by a sharp decrease in fracture stress with increasing temperature.

Typical fracture surfaces in precracked specimens (containing cracks of about 95 micron deep) tested at 1200, 1250 and 1275°C are shown in Fig. 8. The presence of subcritical crack growth at 1275°C is clearly seen in Fig. 8. It is fair to conclude from the study of precracked specimen (Fig. 6) that in hot pressed silicon nitride NC132

*Bars indicate the max. and min. values. Curve is drawn through the lower limits where most of the data points fell. This curve does not represent the data shown in Table 2.

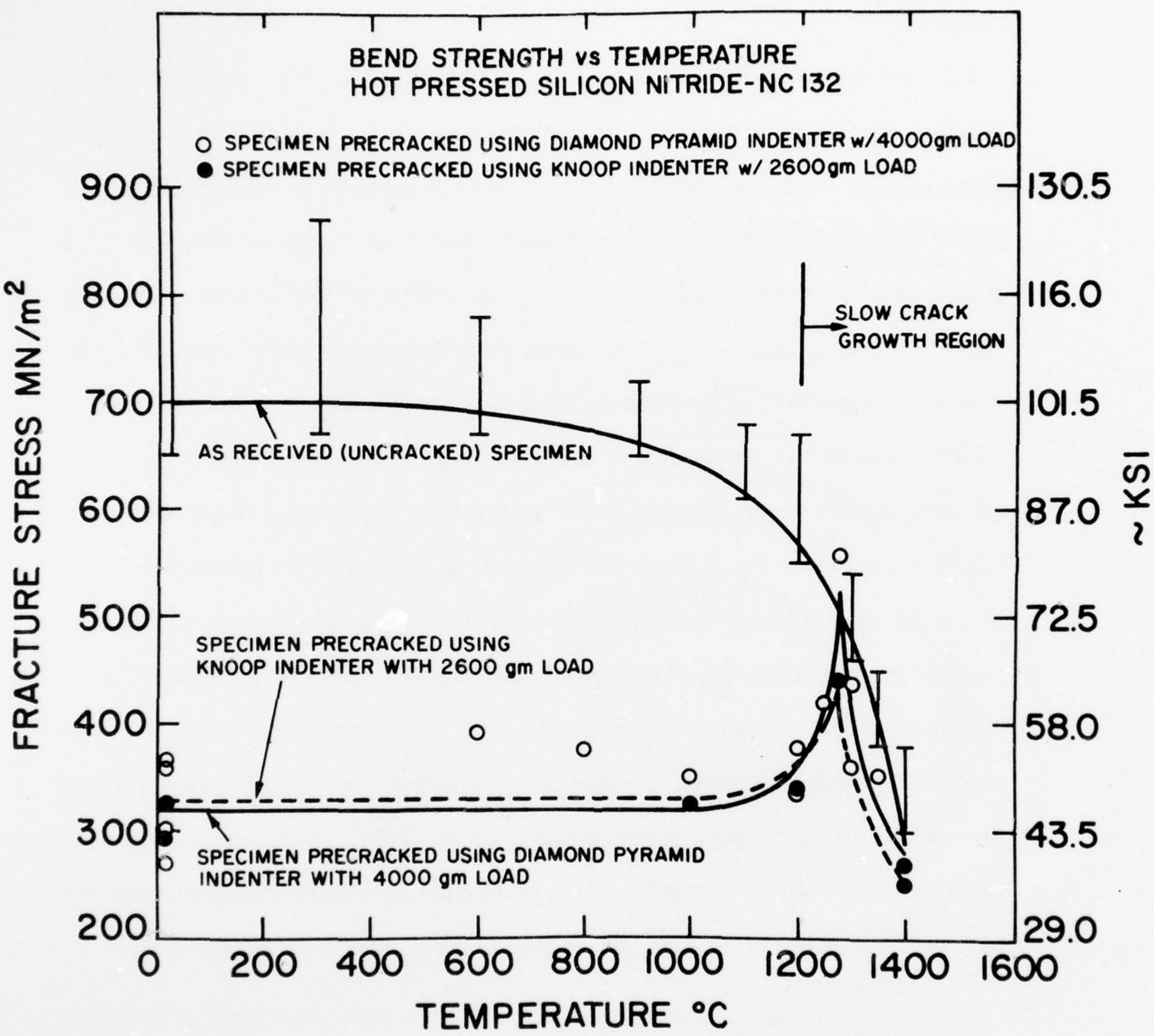
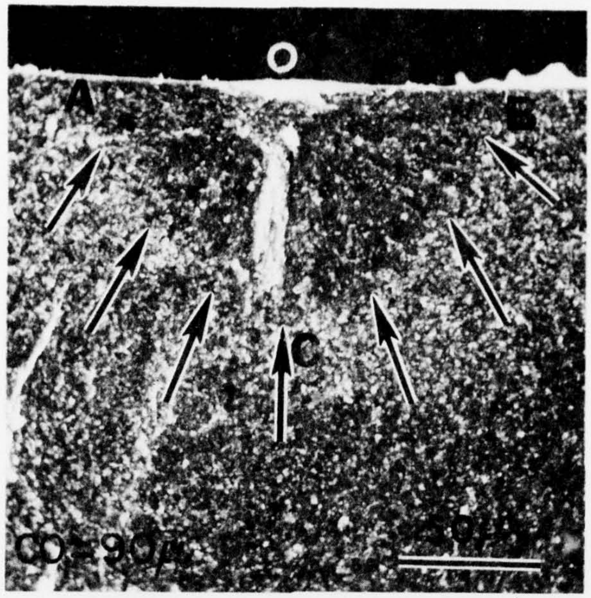
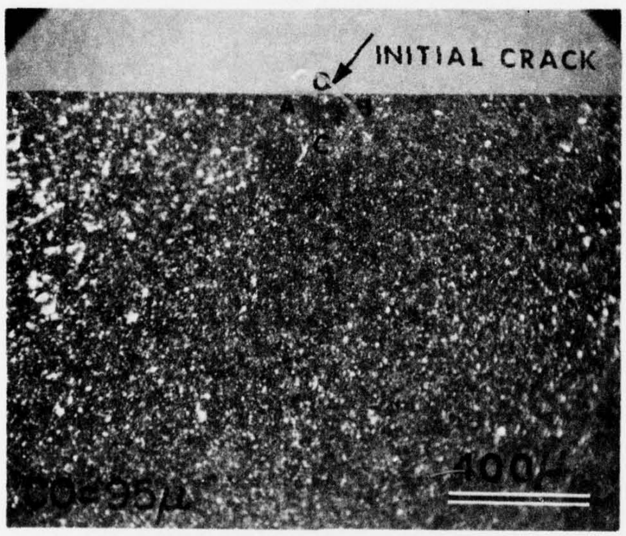


Fig. 6 Effect of temperature on the fracture stress of uncracked and precracked specimens of hot pressed silicon nitride, NC132.



a. 20 °C, 4Kg



b. 1000 °C, 4Kg

Fig. 7 Typical fracture surfaces of specimens tested at 20 and 1000°C. Micrograph shown in (a) is an SEM view while in (b) is an optical view taken in polarized light. Note that the semicircular precracked region, ACB, is clearly visible and free from any tear marks (or cleavage steps).

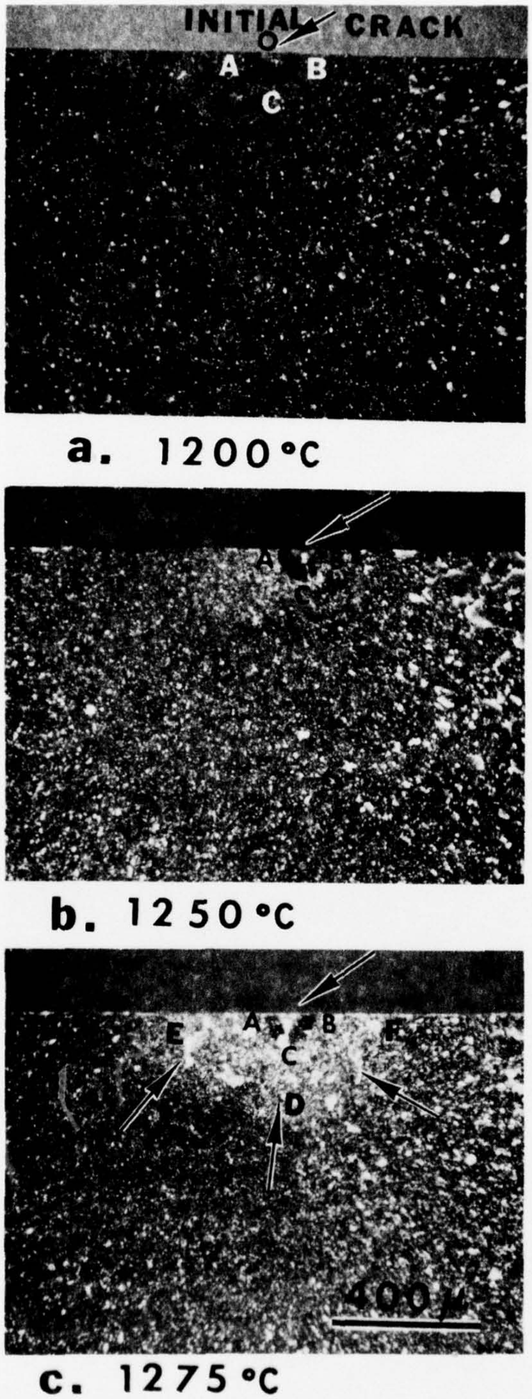


Fig. 8 Typical fracture surfaces of NC132 specimen, precracked with 4 Kg load indentation (approximate depth of crack, CO = 95 micron) and subsequently tested at a machine head speed of 0.005"/min. at various temperatures. Pictures taken with plane polarized light. Note the absence of slow crack growth at 1200 and 1250°C and subsequent appearance of slow crack growth surrounding the initial crack at 1275°C.

subcritical crack growth is evident around 1200°C.* However, it is likely that the presence of subcritical crack growth can occur at much lower temperatures such as 1000-1100°C if the crosshead speed of testing is an order of magnitude slower or the material contains large amounts of impurities such as MgO, Ca, Fe, etc. In conclusion, subcritical crack growth has been observed around 1200°C in hot pressed silicon nitride NC132.

2.5 Effect of Loading Rate on Subcritical Crack Growth in Precracked Specimen

The effect of loading rate (or machine head speed) and temperature (1200, 1300, 1350 and 1400°C) on the presence of subcritical crack growth has been investigated using specimens containing a constant crack size (about 95 $\pm$ 5 micron deep). Increasing the strain rate decreases the amount of subcritical crack growth as expected. Typical fracture surfaces showing successive stages in the development of subcritical crack growth in tests made at 1400°C are shown in Fig. 9. Further work is in progress and results will be analyzed for future reports.

3.0 Flexural Stress Measurements and Rate Testing

An important part of this study is to make a statistical evaluation of the detailed data obtained through flexural stress testing due to the inherent randomness of the strength controlling flaw distribution. Weibull's analysis was employed and the strength controlling properties of materials are defined in terms of the characteristic strength, σ_θ , at which 63.2 percent of the population (specimens) will fail and the Weibull modulus, m, which defines the distribution of flaws in the material. Using simplified Weibull theory, the probability of failure in a brittle material subjected to stress σ is given by

*This conclusion is primarily based on the fact that the speed of testing used in data given in Fig. 6 was 0.005"/min.

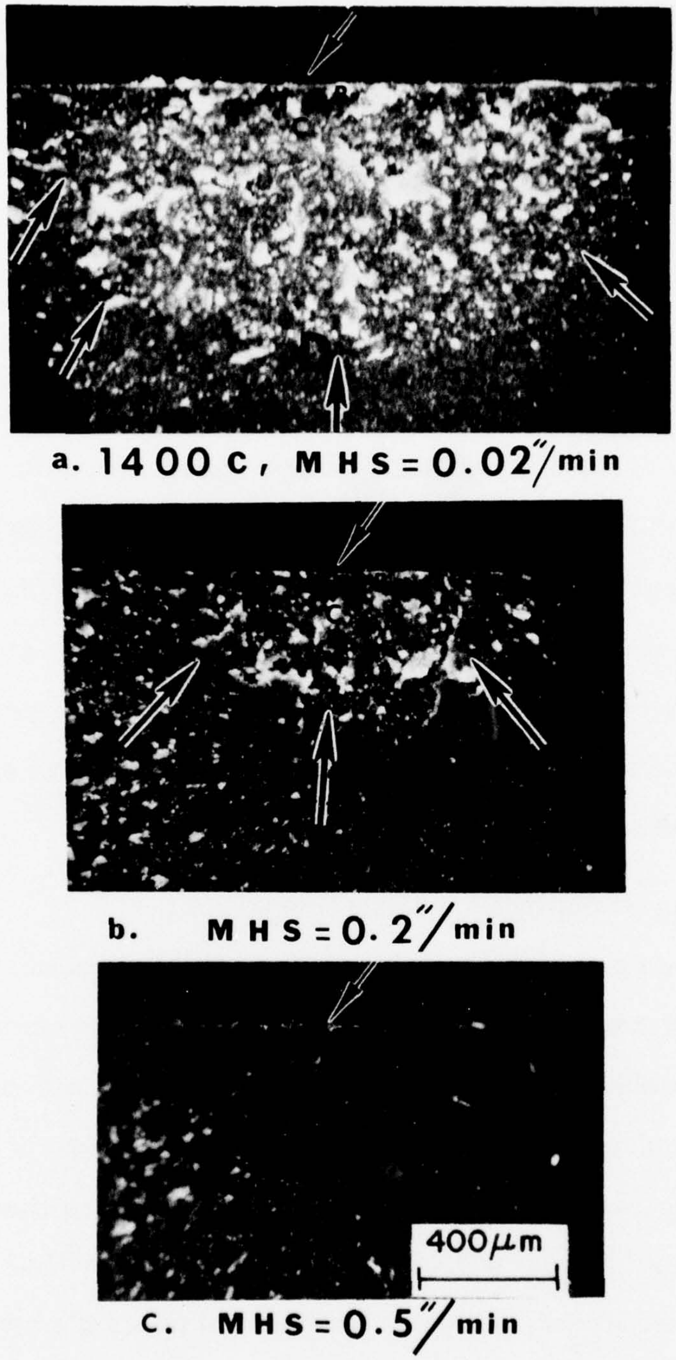


Fig. 9 Successive stages in the slow crack growth of precracked specimen tested at 1400°C as a function of machine head speed. All specimens were precracked with 4 Kg. load indentation. Pictures taken with plane polarized light.

$$P_f = 1 - e^{-\left(\frac{\sigma}{\sigma_\theta}\right)^m} = 1 - R \text{ --------(5)}$$

Where R is the reliability of the structure. The time to failure, t_f, [12] for a given applied stress, σ_A is given by

$$t_f = \frac{\sigma_\theta^{\,n+1}\left(\ln \frac{1}{R}\right)^{\frac{n+1}{m}}}{\sigma_A^{\,n}\ \dot{\sigma}\,(n+1)} \text{ --------(6)}$$

where σ_θ and m were determined at a stressing rate, $\dot{\sigma}$. The parameter 'n' is related to the presence of subcritical crack growth as described below. It is clear from equation (6) that in order to predict the time to failure, t_f, for a structure or component, at a given applied stress, σ_A , and reliability R, three parameters, namely, σ_θ , m and 'n', need to be evaluated at a specified temperature. The maximum-likelihood estimators (MLE) method of statistical analysis was used in determining σ_θ and m using a computer program [13] and the parameter 'n' is determined using the stress rate approach as outlined below. It should be pointed out that this is an alternative, simple and relatively inexpensive approach compared to the Double Torsion method (see Sec. 2.2.1) used to evaluate the subcritical crack growth parameters.

The presence of subcritical crack growth in ceramic materials can be described by the functional relationship between crack velocity, V, and stress intensity factor, K_I, as follows:

$$V = A K_I^{\,n} \text{ - - - - - - - - (7)}$$

where A and 'n' are constants. Flexural stress rate testing [14, 15] is one method for determining the exponent 'n' and utilizes the materials fracture strength as a function of stressing rate as given below:

$$\frac{\sigma_{F_1}}{\sigma_{F_2}} = \left(\frac{\dot{\sigma}_{F_1}}{\dot{\sigma}_{F_2}}\right)^{\frac{1}{n+1}} \text{ - - - - - - - - (8)}$$

where σ_{F_1} is the fracture stress at a stress rate $\dot{\sigma}_{F_1}$, and σ_{F_2} is the fracture stress at a stress rate $\dot{\sigma}_{F_2}$ and 'n' is a constant. The simplicity of this equation (8) is obvious as it only requires two different fracture stresses measured at two different stress rates in order to determine the slow crack growth exponent 'n' at a given temperature. Equation (8) can also be represented [16] in the following manner

$$\log \sigma_{F_1} = \log D + \frac{1}{n+1} \log \dot{\sigma}_{F_1} \text{ - - - - - - - - (9)}$$

where D is a constant and a log-log plot of σ_F vs stressing rate $\dot{\sigma}_F$ would yield a linear relationship and from the slopes of the plot, the parameter 'n' can be determined.

3.1 Sample Preparation and Testing

Three 6 in. x 6 in. billets of hot pressed silicon nitride NC-132, were used to make MOR test specimens, two of which were 1 in. thick and the third one was 0.75 in. thick. MOR bars of dimensions 1.25 in. long x 0.25 in. wide x 0.125 in. thick were cut such that the tensile face was perpendicular to the hot pressing direction (strong direction) Fig. 10. The machining process is described in Table 6. The Ford HP-SN MOR specimens were cut from the tapered hubs of duodensity Si_3N_4 rotors, representative of those fabricated during 1977. They were fabricated using Kawacki Beryllium Company CP 85 Si_3N_4 powder and 3.5 weight percent MgO as a sintering aid. The mixture was hot pressed at a pressure of 1000 psi and at a temperature of 1700°C with a hold of 3 hrs., following a standardized procedure. Material for this program came from rotors which generally had been rejected due to defects within the reaction sintered Si_3N_4 blade zone and were therefore unacceptable for rig or engine testing. After removal of the blade material, standard bars were cut from the remaining HPSN hub material in a similar fashion to that shown in Fig. 10. For most of the test temperatures, bars cut from 6 rotors were used. Specimens from a seventh rotor were added for the 1204°C tests.

All specimens were tested in four-point bending in an Instron machine (model 1125) using a specially built self aligning ceramic fixture, Fig. 11, made from hot pressed SiC. The outer and inner knife edges were 0.75 in. and 0.38 in. span respectively. The high temperature bend tests were conducted in air in an Instron machine equipped with a rapid temperature furnace.* Initially, bend bar specimens of both types of material (hot pressed silicon nitride NC-132) and 3.5% MgO FHPSN) were tested in bending at a crosshead speed of 0.02 in./min. (0.5 mm/min). Bend bar specimens were tested at higher temperatures using two additional crosshead speeds as shown in Tables 2 and 3 for both types of material, NC-132 and FHPSN, respectively.

*CM Inc., High Temperature Furnaces, Bloomfield, N.J.

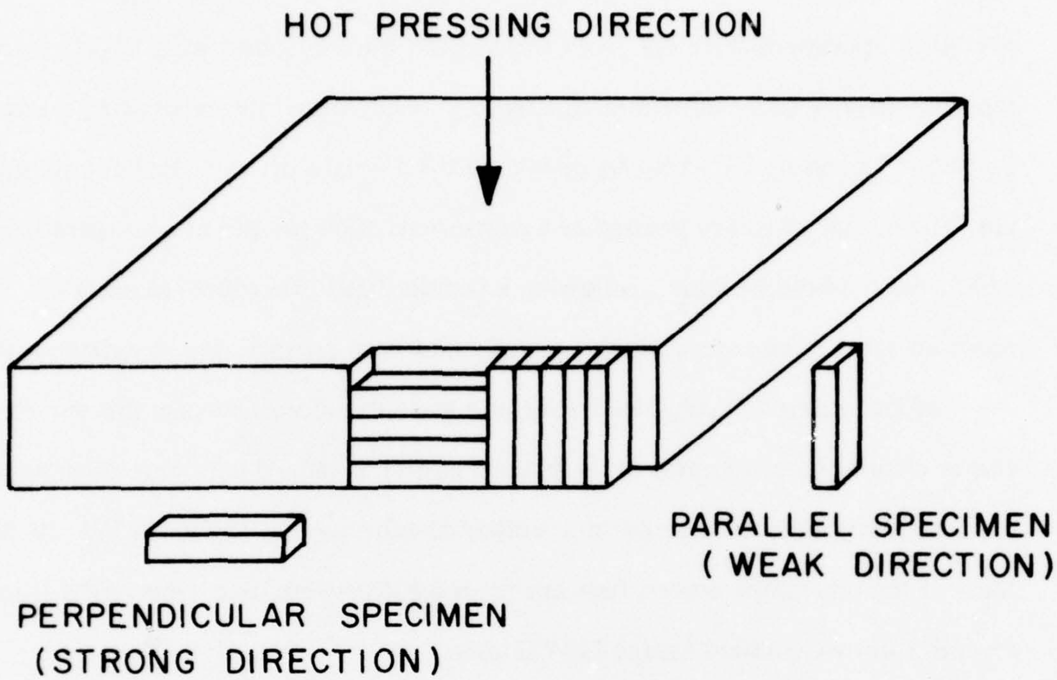


Fig. 10 Schematic representation of specimen layout from the hot pressed silicon nitride billet showing strong and weak directions.

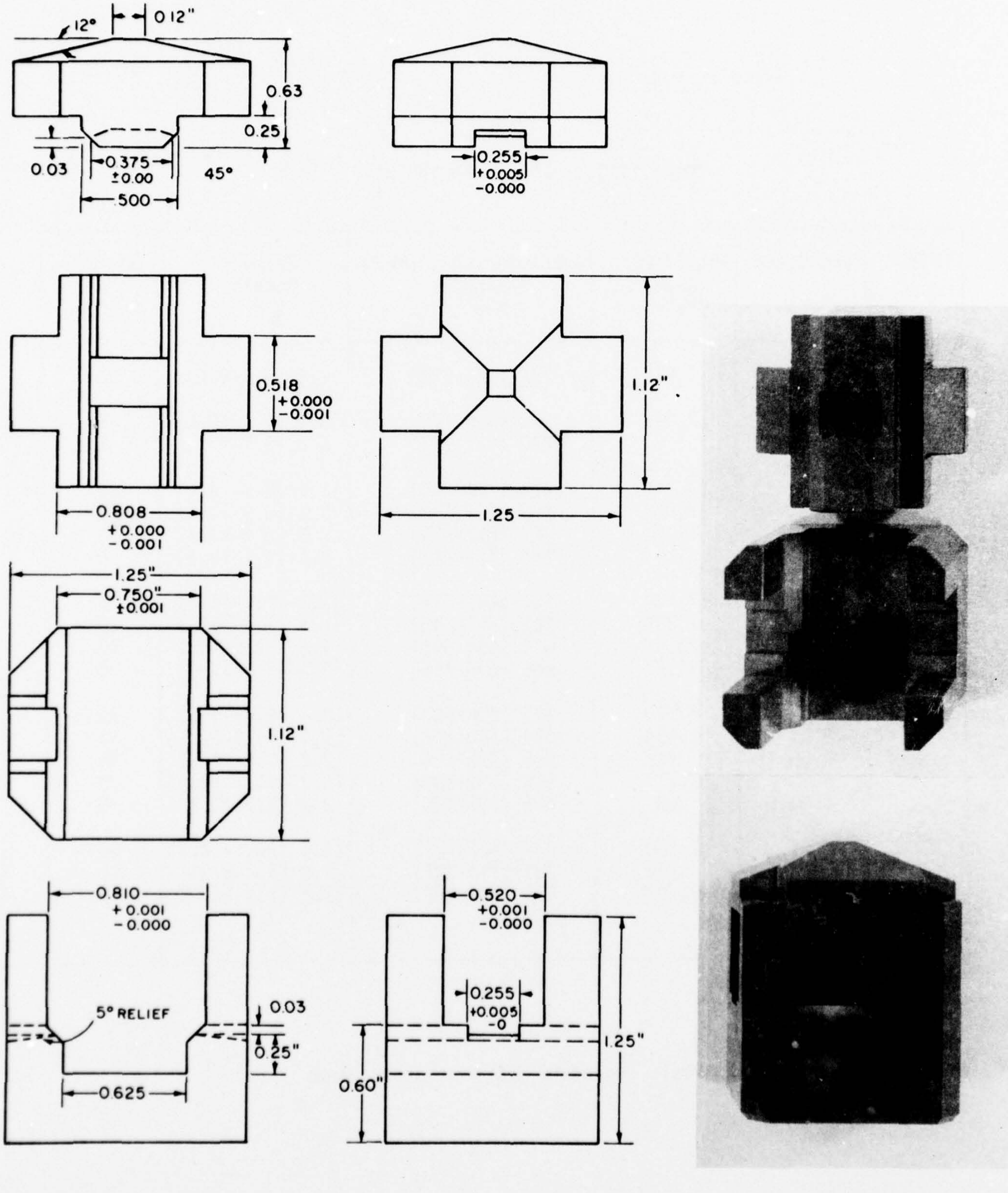


Fig. 11 Details of the self aligning ceramic fixture used in high temperature bend tests.

TABLE 2

(MOR) STRESS DATA OF NORTON NC-132 HPSN

Temp.	Head Speed mm/min	Avg. Stress Rate MPa/min	Characteristic (MOR) Strength MPa	Weibull Modulas m	No. Of Samples
Room	0.5	1,870	787 (748-828)	6.5 (4.9-8.0)	30
704°C	0.5	2,090	730 (686-778)	8.0 (5.1-10.4)	15
	0.0005	1.8	730 (607-901)	6.0 (2.1-8.7)	5
871°C	5.0	19,740	728 (708-750)	11.6 (8.7-14.1)	30
	0.5	2,000	759 (732-788)	9.2 (6.9-11.2)	30
	0.005	21.9	727 (697-759)	7.9 (5.9-9.7)	30
	0.0005	1.8	818 (719-947)	8.5 (3.1-12.5)	5
1038°C	5.0	17,100	725 (697-754)	12.6 (8.1-16.4)	15
	0.5	1,880	725 (711-740)	16.5 (12.4-20.2)	30
	0.005	22.5	679 (664-695)	14.5 (10.8-17.7)	30
	0.0005	1.5	666 (596-756)	9.9 (3.6-14.5)	5
1204°C	5.0	18,120	665 (648-683)	13.0 (9.8-15.9)	30
	0.5	1,830	688 (680-697)	25.8 (19.4-31.5)	30
	0.05	163	609 (593-626)	12.2 (9.2-14.9)	30
	0.005	21.1	544 (532-556)	15.7 (11.7-19.2)	30
	0.0005	.8	485 (421-566)	8.0 (2.9-11.7)	5
1371°C	5.0	11,900	411 (402-421)	14.3 (10.8-17.5)	30
	0.5	959	363 (347-381)	7.0 (5.2-8.5)	30
	0.05	91.0	313 (299-327)	11.2 (7.2-14.6)	15

Note: Numbers in parenthesis represent 90% confidence bands

TABLE 3

(MOR) STRESS DATA OF FORD 3.5%MgO HPSN

Temp.	Head Speed mm/min	Avg. Stress Rate MPa/min	Characteristic (MOR) Strength MPa	Weibull Modulas m	No. Of Samples
Room	0.5	1,770	668 (646-691)	10.0 (7.5-12.2)	30
$704^{o}C$	0.5	1,860	697 (662-735)	6.4 (4.8-7.8)	30
$871^{o}C$	0.5	1,770	591 (552-624)	4.8 (3.6-5.9)	30
	0.05	207	597 (556-647)	6.7 (4.3-8.7)	15
	0.005	20	579 (544-617)	5.3 (4.0-6.5)	30
$1038^{o}C$	0.5	1,740	548 (529-569)	9.2 (6.8-11.2)	30
	0.05	196	596 (572-621)	15.2 (8.8-21.5)	10
	0.005	19	531 (517-546)	12.4 (9.3-15.1)	30
$1204^{o}C$	0.5	1,580	481 (455-509)	5.5 (4.2-6.6)	35
	0.05	172	438 (411-467)	4.8 (3.7-5.8)	35
	0.005	17.5	368 (356-381)	9.0 (6.9-10.8)	35
$1371^{o}C$	0.5	560	254 (244-263)	7.9 (5.9-9.7)	29

Note: Numbers in parenthesis represent 90% confidence bands

3.2 Results and Discussion

Considerable time, care and effort have been spent in obtaining a statistical data base for two potential ceramic turbine engine materials, hot pressed silicon nitride NC-132 and 3.5% MgO FHPSN. All measurements were made using the four-point bend test (Flexural Stress) because of its simplicity and the relatively inexpensive nature of the test compared to other tests such as uniaxial tension, indentation induced cracking and double torsion, etc. A total of 425 and 339 bend bar specimens for NC-132 and FHPSN materials respectively were tested as a function of temperature and stressing rate in order to apply statistical analysis procedures in predicting materials behavior related primarily to the presence of subcritical crack growth. Complete data are given in Tables 2 and 3.

The temperature dependence of the fracture stress as a function of stressing rate on a log-log scale (using equations 8 and 9, Sec. 3) are shown in Figs. 12 and 13 for NC-132 and FHPSN materials, respectively. Up to about 900°C, the fracture stress, σ_F, is independent of the stressing rate $\dot{\sigma}_F$, for both materials as shown by the horizontal line on the log-log plot, Figs. 12 and 13, indicating no evidence of subcritical crack growth (SCG). In general, metallographic examination supported this view. When the temperature is increased to 1038°C, slight deviation from the horizontal line in the log σ_F vs log $\dot{\sigma}_F$ plot for NC-132 was noticed, Fig. 12, indicating the presence of SCG, though metallographic examination of the fracture faces did not reveal clearly the presence of it on a macroscopic scale (10X). However, it is possible that some SCG may have occurred and the absence of it in metallographic examination was simply due to the fact that the broken test pieces were not just two halves but several and the inability to trace the exact initiation site. An approximate calculated value of 'n' using equation (8) was 72 for NC-132 at 1038°C. This relatively large value of 'n' also indicates the absence of SCG at this

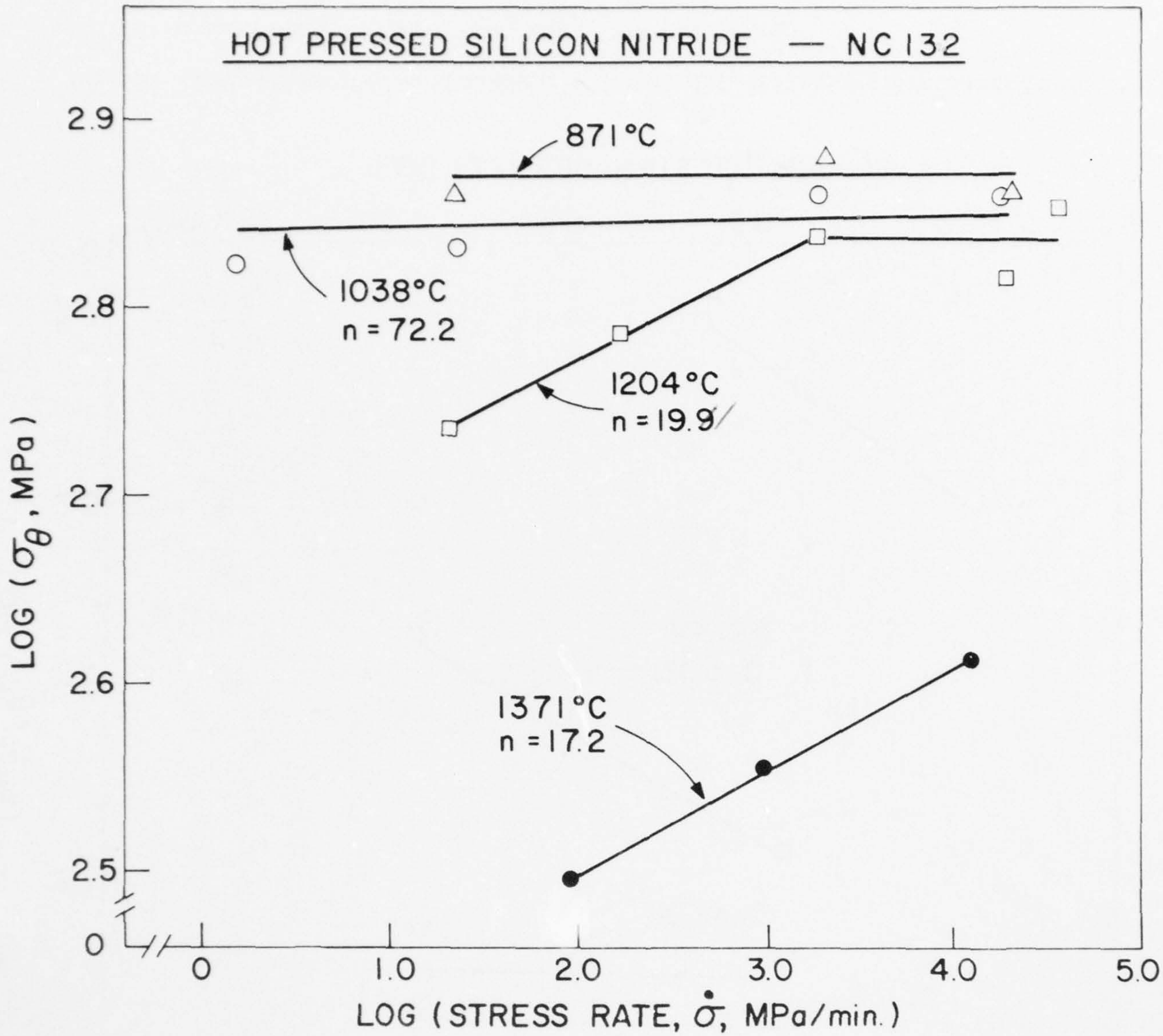


Fig. 12 Log-log plots of flexural strength vs stressing rate at various temperatures for NC132 Si_3N_4.

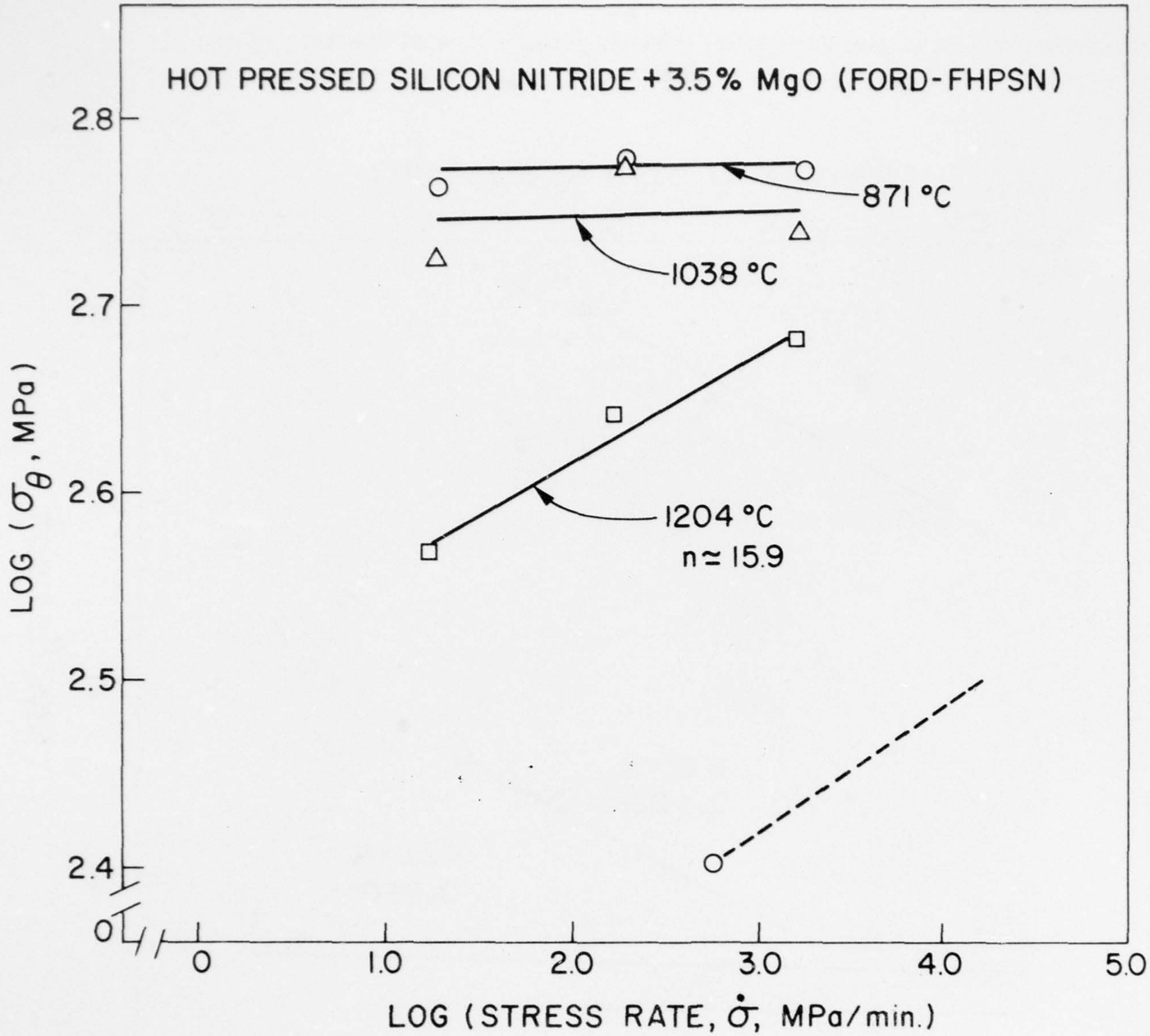


Fig. 13 Log-log plots of flexural strength vs stressing rate at various temperatures for hot pressed silicon nitride + 3.5% MgO (FHPSN) material.

temperature. The FHPSN showed a horizontal line, Fig. 13, also suggesting no SCG at 1038°C. At higher temperatures such as 1204°C and 1371°C, the log-log plot showed a clear deviation from the horizontal line for both types of materials indicating presence of SCG. Values of 'n' as determined using the empirical relationship, equation (8), were 19.1 and 17.2 for NC-132 and 15.9 and 15.9* for FHPSN at 1204°C and 1371°C respectively. For low stressing rates, metallographic examination supported the presence of SCG in accordance with the Figs. 12 and 13. It should be noted that there is not a substantial variation in 'n' either at temperatures >1200°C or with composition. However, the reader is cautioned to the fact that the data and results presented in Tables 2, 3 and Figs. 12, 13, respectively, are preliminary, and future plans include more careful examination of the complete data.

In conclusion, using the flexural stress rate method, values of σ_θ , m and 'n' have been measured from room temperature (20°C) to 1375°C. Using these parameters, the time to failure, t_f, for a given applied stress at a given temperature will be evaluated and compared with stress-rupture tests.

*Based only on one set of data points.

4.0 Stress Rupture Testing

In this study, stress rupture testing will be used as one of the methods to determine the time dependent behavior of hot pressed silicon nitride NC-132 and FHPSN. Two approaches for stress rupture testing have been adopted, namely, tensile stress rupture and flexural stress rupture testing. The former is a preferred method because of uniformity of stress in the cross-section of the test specimen. However, it should be emphasized that testing in tension of brittle, notch-sensitive ceramic materials is a complex problem and the complexities increase with high temperature testing. The flexural stress rupture method will use standard bend bar specimens which will be subjected to various stress levels and time to failure at a specified temperature will be noted. This method is relatively simple and inexpensive compared to tension testing.

4.1 Load Train Design and Development

The tensile stress specimen consists of a simple rectangular geometry with a narrow cross-section in gage length and large radii at the shoulders, necessitated by the sensitivity of ceramic materials to stress concentrations. A schematic of the specimen together with other attachments in the load train assembly is shown in Fig. 14. The gage section is 0.5 in. long x 0.125 in. wide and 0.125 in. thick. The specimen is retained in two slotted SiC holders by large SiC pins. The SiC holders are retained in water cooled metal adaptors which in turn are attached to the standard Satec machine head which includes crossed (90°) knife edges. The assembly procedure includes hanging the load train parts from the Satec head as influenced by gravity. At this point, the lower Satec cross-arm is lowered to snub the train in this position.

Preliminary dry runs at room temperature using a steel specimen with strain gages attached on all four sides have been made to measure bending stresses in the system. Presently, bending stresses of the order of 10% with full load are associated in the system. Efforts will be made to reduce these to the order of 3 to 5% at full load.

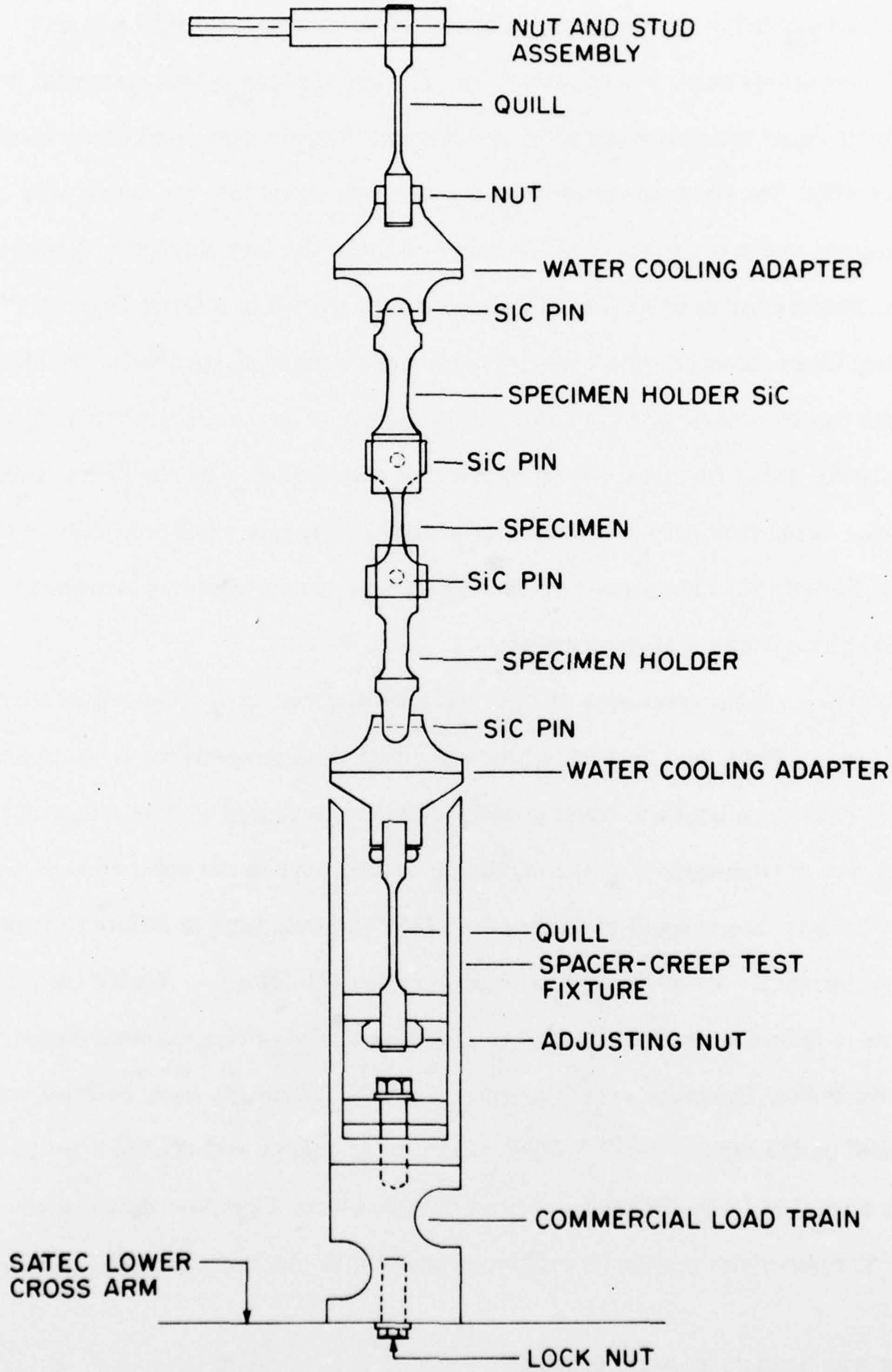


Fig. 14 Schematic representation of the load train assembly used for tensile stress rupture testing at high temperatures.

4.1.1 Load Train Instrumentation

The load applied to the specimen is transferred to an Interface* load cell with a 0-1000 lb. sensitivity and a 1% possible error. The applied load is then converted into a millivolt signal which is received by a Daytronic** strain gage conditioner Model number 9171. The conditioner converts the millivolt signal into the actual load (in lbs.) applied to the specimen. This load value is indicated on a Daytronic digital indicator Model number 9530. The MV signal is then relayed to a Doric Digitrend*** 200 Strip Chart recorder, which subsequently records the load in pounds. The Doric recorder can be programmed to record continuously or at specific intervals. In addition to recording the load, the temperature is also recorded by the Doric, which receives a signal from a Pt-Rhodium thermocouple placed in close proximity to the sample. Figure 15 is a block diagram of the load sensing and recording instruments.

4.2 Flexural Stress Rupture Measurements

Standard bend bar specimens of both types of material were subjected to a constant load at 1204°C and time to failure was noted. This temperature is particulary suitable because subcritical crack growth (SCG) is evidenced at this temperature (Sec. 3) and furthermore, it is the maximum temperature in the outer ceramic turbine rotor hub. Stress levels were chosen to limit the total time to failure to within 100 hrs. Specimens from NC-132 were subjected to 415 MPa (~ 60,200 psi) and the time to failure varied from 0.5 hr. to 18 hrs. in a total of 10 specimens from two different billets. Complete data is given in Table 4. Specimens from FHPSN were subjected to 256 MPa (~ 37,100 psi) and time to failure varied from 1 hr. to 95 hrs. in a total of 10 specimens from two different billets. Complete data is given in Table 5. From these preliminary measurements, it is too early to make any con-

* Interface, Scottsdale, Arizona 85260
**Daytronic, Miamisburg, Ohio 45342
***Doric, San Diego, California 92123

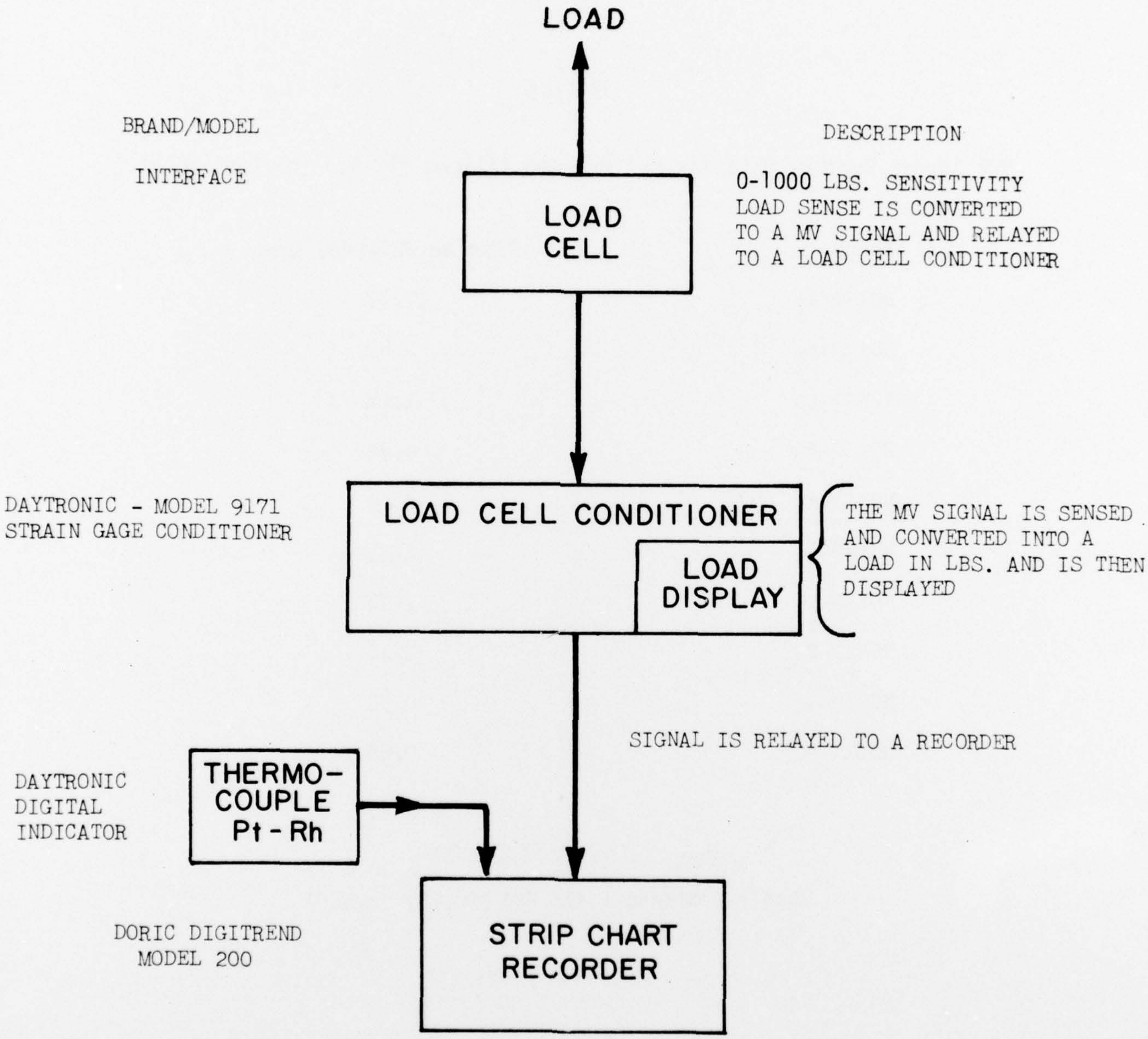


Fig. 15 A block diagram view of the load sensing and recording equipment associated with load train assembly.

TABLE 4

MOR Stress Rupture Data for Hot Pressed Silicon Nitride (Norton, NC 132)

Sample	Time to Failure, Hrs.
NC132-A_1	17.97
NC132-B_1	2.42
NC132-A_2	1.15
NC132-B_2	0.75
NC132-A_3	4.16
NC132-B_3	1.33
NC132-A_4	1.75
NC132-B_4	2.16
NC132-A_5	0.55
NC132-B_5	0.58

Temp. 1204°C (2200°F)

Applied Stress ≈ 415 MPa (60,187 lb/in^2)

TABLE 5

MOR Stress Rupture Data for Hot-Pressed Silicon Nitride + 3.5% MgO (FHPSN)

Sample	Time to Failure, Hrs.
1215 A_1	2.87
1216 B_1	9.07
1215 A_2	9.53
1216 B_2	63.12
1215 A_3	7.73
1216 B_3	18.08
1215 A_4	7.73
1216 B_4	83.58
1215 A_5	1.08
1216 B_5	95.70

Temp. 1204°C (2200°F)

Applied Stress ≃ 256 MPa (37,128 lbs/in^2)

TABLE 6

Standard Bar Preparation Procedure

Slicing

Wheel Spec.	Resin 120 grit
Wheel Speed	5000 - 6000 SFPM
Downfeed	.0005" - .001"
Table Speed	100 - 140 inches/min

Rough Grind (when there is enough material allowed)

Wheel Spec.	Resin 100 grit
Wheel Speed	5000 - 6000 SFPM
Downfeed	.0015" - .002" inches/pass
Crossfeed	1/8 - 1/4 inches/pass
Table Feed	300 - 400 inches/pass

Intermediate Grind

Wheel Spec.	Resin 150 grit
Wheel Speed	5000 - 6000 SFPM
Downfeed	.008" - .0015"
Crossfeed	1/8 - 1/4
Table Speed	200 inches/min

Finish Grind

Wheel Spec.	Resin 280 grit
Wheel Speed	5000 - 6000 SFPM
Downfeed	.0003" - .0005"
Table Speed	100 - 140 inches/min

All final grinding done parallel to the long axis of the specimen.

All edges bevelled .005" - .010" by lapping in longitudinal direction.

clusive remarks.

A detailed and carefully planned work will be carried out using the flexure stress rupture method on both types of material in order to predict the time to failure for given applied stresses for a given temperature and correlated with those obtained from stress rupture tension tests. Finally, efforts will be made to draw some SPT (Stress/Probability of Failure/Time) diagrams which a design engineer could use to obtain the necessary information.

5.0 Proof Test Methodology

One of the objectives of the Ceramic Life Prediction/Proof Test Methodology Program is to investigate whether the survival probability of a ceramic structure subjected to strength degradation resulting from subcritical crack growth (S.C.G.) can be enhanced by the prior application of a proof test. It is postulated that the population of flaws that control the strength of a ceramic structure can be effectively truncated by loading the structure to a predetermined stress level and eliminating in the process "defective" pieces with "excessive" flaws. The investigation will be carried out on simply stressed and geometrically simple test specimens, mainly for economic reasons, so that meaningful statistical sampling is assured within economic resources available. Proof testing will be limited initially to room temperature under the assumption that the crack population remains invariant with temperature.

Analytical relations that will be used in studying the effects of proof tests are derived next.

5.1 Analytical Procedure

The strength of a ceramic structure, such as a test bar is conveniently expressed in terms of Weibull parameters [17]

$$(S)_{t=o} = S_{\theta\, t=o} \left(\ln \frac{1}{R} \right)^{\frac{1}{m}} \quad \text{--------} (10)$$

S_θ being the characteristic strength of the structure (a function of Weibull parameter S_o and the stress distribution as expressed by so called effective volume), and 'm' the shape parameter, popularly called Weibull slope. R is the reliability level associated with the strength level S.

In the presence of subcritical crack growth, the strength and its distribution change with time [18]

$$(S)^{n+1}_{t=t_f} = S^{n+1}_{t=o} - \sigma^{n}(n+1)\, t_f \dot{\sigma} \quad \text{--------(11)}$$

t_f – time at load

n – crack propagation exponent

σ – applied stress

$\dot{\sigma}$ – stress rate at which the characteristic strength S_θ was measured or computed

Probability of survival under the applied stress σ also changes with time.

$$R_{t=t_f} = \exp\left\{ - \left[\left(\ln \frac{1}{R}\right)_{t=o} \left(1 + \frac{t_f (n+1)\, \dot{\sigma}}{\sigma}\right)^{\frac{m}{n+1}} \right] \right\} \quad \text{--------(12)}$$

The strength of a ceramic test bar and thus its survival probability under stress σ can be enhanced by the application of a proof load which is characterized by the proof stress magnitude σ_p and proof stress distribution (S_{θ_p}). In tests contemplated in this investigation, it is fair to assume that the stress distributions corresponding to σ and σ_p will be identical so that

$$S_{\theta_p} = S_\theta \quad \text{--------(13)}$$

Thus the initial strength of a ceramic test component after the proof test becomes

$$(S)^{m}_{t=o} = S^{m}_{\theta\, t=o} \left(\ln \frac{1}{Ra}\right) + \sigma_p^{\,m} \quad \text{------(14)}$$

R_a being the probability associated with the strength level S of the proof tested component.

Again in the presence of S.C.G. both the strength and reliability degrade with time

$$(S)^{n+1}_{t=t_f} = \left[S^{m}_{\theta\, t=o} \left(\ln \frac{1}{Ra}\right) + \sigma_p^{\,m} \right]^{\frac{n+1}{m}} - \sigma^{n} t_f (n+1) \dot{\sigma} \quad \text{--------(15)}$$

and

$$\left(R_a\right)_{t=t_f} = \exp\left\{-\left[\left(\ln\frac{1}{R}\right)_{t=o}\left(1+\frac{t_f(n+1)\dot{\sigma}}{\sigma}\right)^{\frac{m}{n+1}} - \left(\frac{\sigma_p}{S_{\theta p}}\right)^m\right]\right\} \qquad \text{--------(16)}$$

The magnitude of the proof test depends entirely on the level of reliability one is looking for in a component. For simplicity let us assume that we would like to negate the deleterious effects of SCG, that is we want the reliability of a proof tested component after lapse of time t_f to be equal the initial reliability of an unproof tested component, i.e.

$$\left(R_a\right)_{t=t_f} = R_{t=o}$$

In which case

$$\sigma_p = \sigma\left[\left\{\left(1+\frac{t_f(n+1)\dot{\sigma}}{\sigma}\right)^{\frac{m}{n+1}} - 1\right\}^{\frac{1}{m}}\right] \qquad \text{--------(17)}$$

and since generally

$$\frac{t_f(n+1)\dot{\sigma}}{\sigma} \gg 1 \qquad \text{--------(18)}$$

we have

$$\sigma_p = \sigma^{\frac{n}{n+1}}\left[t_f(n+1)\dot{\sigma}\right]^{\frac{1}{n+1}} \qquad \text{--------(19)}$$

or

$$\frac{\sigma_p}{\sigma} = \left(\frac{t_f(n+1)\dot{\sigma}}{\sigma}\right)^{\frac{1}{n+1}} \qquad \text{--------(20)}$$

The magnitude of the proof stress as we see from eq. 20, is related inversely to the level of the operating stress in which one is proof testing. Thus the lower the operating stress, the larger the initial reliability the larger the proof load required to negate the degradation incurred as the result of SCG.

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This program consists of a basic study using two potential high temperature ceramic materials, namely, hot pressed silicon nitride NC132 (Norton) and hot pressed silicon nitride made with 3.5 wt. percent MgO (Ford material) to determine the statistical and time dependent strength characteristics related to the presence of subcritical crack growth. The data will be used to correlate analytical ceramic life prediction and possibly verify with experimental results obtained from simple tensile stress rupture tests.

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